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Makrofol nuclear track filters : effect of surfactant on electrolytically controlled etching

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Abstract : Surfactants decrease surface tension which, in turn, increases the wettability. Anionic and non-ionic surfactants have been in a use as small additives to the main chemical etchants for etching of solid state nuclear track detectors. Addition of a non-degrading surfactant to the etchant improves the etching conditions by increasing wetting and removal of the passivated surface layers formed by chemical reaction products. The wettability of a surfactant may be different for different polymers, under given conditions. In this paper, results obtained from the Sessile drop experiment are described which provide information as to the usefulness of different commercially available surfactants for increasing wettability and hence etch-rates in N and KG type Makrofol. Effects of addition of surfactants to the main electrolyte (etchant) used in electrolytically controlled etching of Makrofol nuclear track filters are also discussed and explained.

Keywords : Surfactants, wettability, electolytically controlled etching, pore radius, break-through time, nuclear track filters.

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1. Introduction

The Nuclear Track Filters (NTFs) are produced by irradiation of suitable dielectric materials with energetic heavy ions followed by the chemical etching (say electrolytically controlled etching) with suitable etchant. Electrolytically controlled etching is convenient in submicron pore size range (Chakarvarti *et al* 1986) and this method has been applied for production of NTFs. Surfactants added to liquids modify their mobility on solid surfaces. The addition of surfactants to etchant decreases surface tension and hence increases wettability. The wettability of a surfactant depends upon the concentration and nature of the surfactant. It also depends upon the material surface to be used for wettability test. The studies on the effect of surface active agents, and contact angle hysteresis using Sessile drop experiments (Singh 1986, Padmanabhan and Bose 1988) have bearing upon the surface tension and wettability properties. In such experiments a drop of critical volume of a

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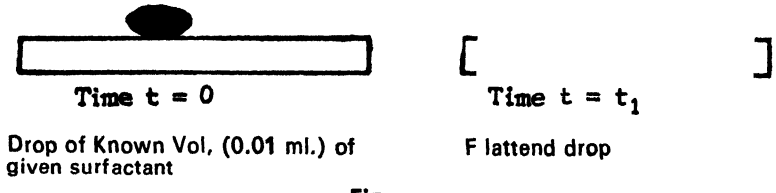
given liquid is observed for its movement on nonhorizontal solid surface. In the present work, we have studied the rate of spread of a given volume of a surfactant solution on a horizontal surface of a detector foil as a measure of wettability. After the drop is placed on the horizontal sheet, it spreads to an equilibrium state (Nguyen *et al* 1987) and the rate would depend upon factors like nature of liquid, critical advancing contact angle between the drop and solid surface at the contact drop volume, wettability, etc.

2. Experimental

Makrofol N (60 μm, Bayer AG, Lever Kusen, FRG) and CN-85 (Kodak-Pathe', 100 μm, France) were the materials used as substrates for the wettability study. The spread of a given drop on the polymer sheet is shown in Figure 1.



Figure

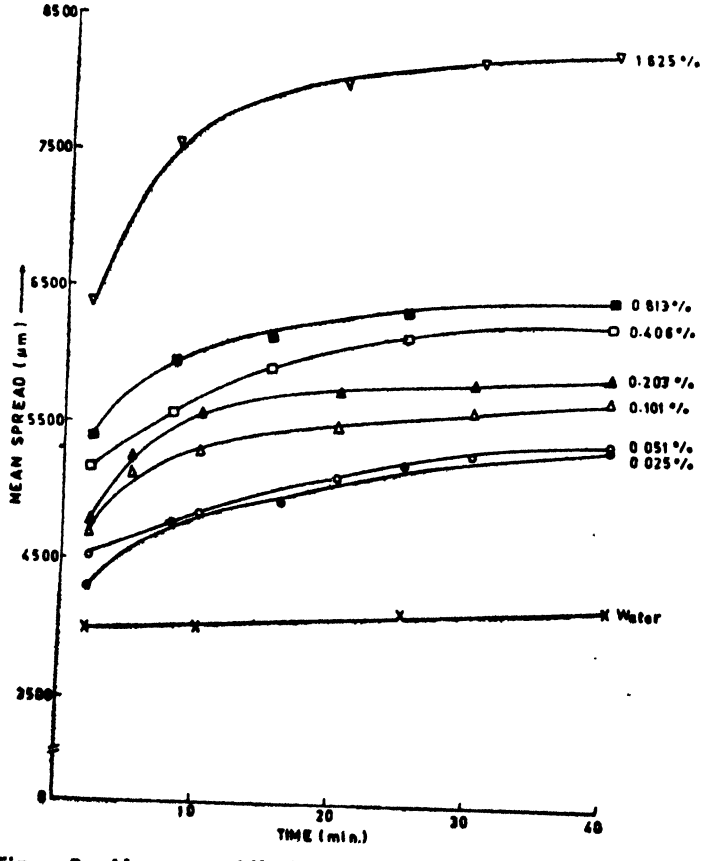


Figure 2. Mean spread Vs time curves for Ezee surfactant on Makrofol N.

A drop of known volume was placed on the polymer sheet. At t=3 sec., mean spread of the drop was measured under the optical microscope. The same set, then, was repeated for longer time duration. Two surfactants-Ezee (Godrej, Bombay) and Genteel (Swastik, Bombay) were added in distilled water in different concentration. For studying the effect of surfactants on the electrolytically controlled etching of NFTs, Ezee surfactant was added in 6N NaOH in varying concentrations.

3. Results and discussion

Figures 2-4 show the variation in spread (μm) with time for different polymer sheets using different concentration of surfactants. It is clear from Figures 2 and 3

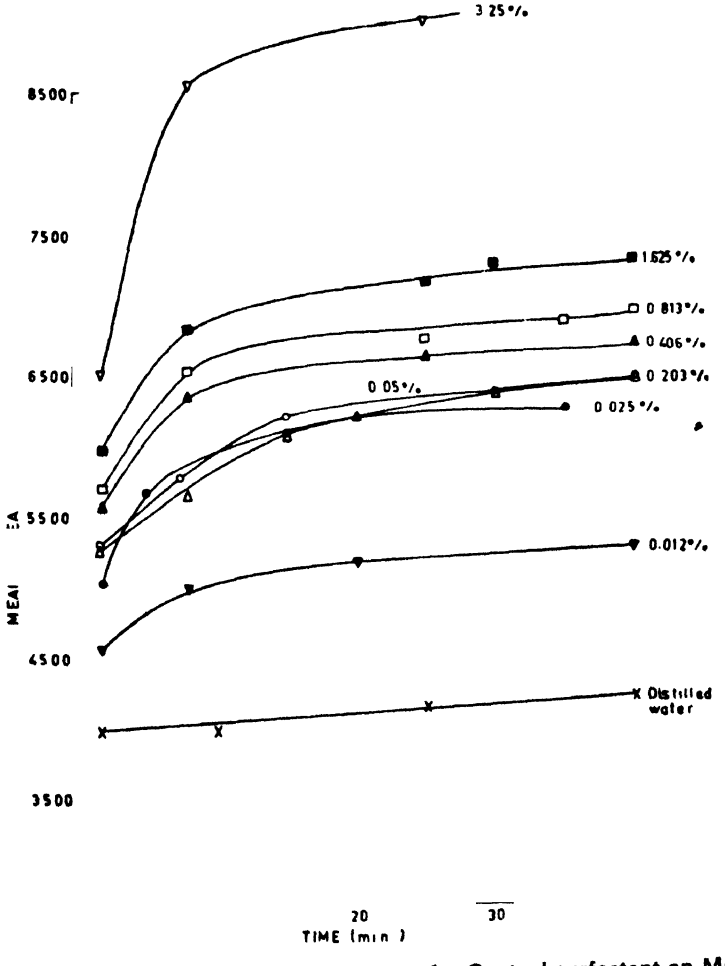


Figure 3. Mean spread Vs time curves for Genteel surfactant on Makrofol.

that the wettability of Genteel is less than that of Ezee. In case of Ezee, we can measure the wettability upto the 1.63% concentration, but for Genteel it can be

measured upto 3.25% accurately. To show the dependence of wettability on the polymer surface, we can compare Figures 3 and 4. One finds that the curve for 3.25% for Makrofol is very steep initially, while in case of CN-85, it is systematic and linear.

For the break-through study, the etching chamber was used. Samples of 1 cm × 1 cm (^{238}U ion with specific energy of 14 MeV/n and with fluence of

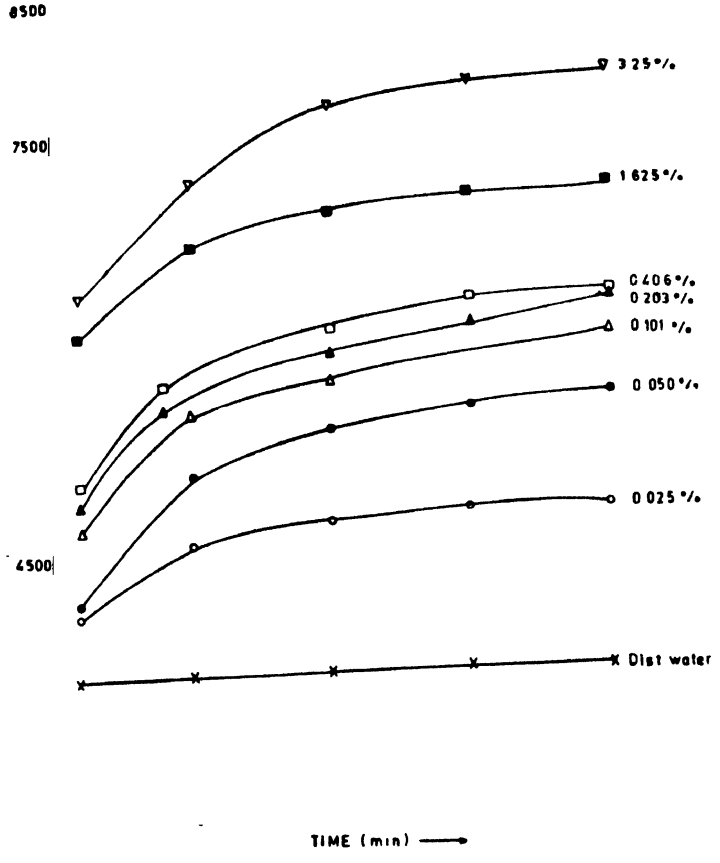


Figure 4. Mean spread Vs time curves for Genteel surfactant on CN-85 (Kodak).

$10^5/cm^2$ were normally incident) were used and the break-through time – the time when two growing cones from opposite sides of the foil meet in the centre of the foil. The pore radius is given by relation (Mittmann *et al* 1981).

$$r(t)=\frac{(l_0-2V_b t)}{R_F \pi N}$$

where V_b is the bulk etch rate of the foil material. The value of N is calculated by knowing the fluence of incident ions on the surfact area of foil and R_F can be found by comparing potential difference V_R across a standard resistance R and potential difference V_F across the foil giving $R_F = R \times V_F/V_R$.

The variation of pore radius with etch time, with and without surfactant added to the main etchant is shown in Figure 5. In the initial moment of etching, the rate of V_t is decreased when compared with the ones etched without using surfactant. This is shown by the increase in break-through time (t_D). As the concentration of surfactant used is increased, the t_D value also shows the increasing

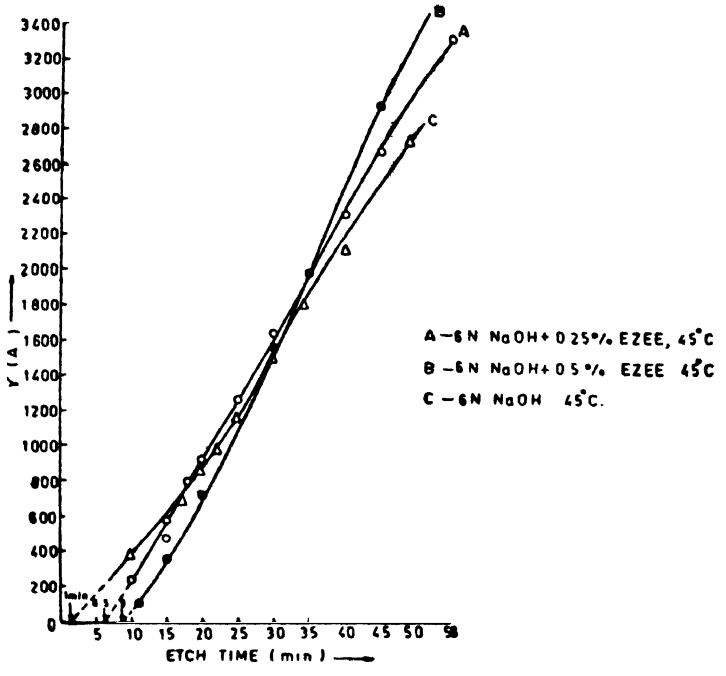


Figure 5. Pore radius vs etch time profiles for different concentration of surfactant.

trend. However, there is a cross-over after time $t > t_D$ (say $t = 40$ min) where the trend reverses and the track etch velocity increases with the concentration of surfactant added. There is an increase in pore radius by 9.5% with 0.25% surfactant and by 21% by 0.5% surfactant.

Therefore the addition of surfactants to the etchant improve the etching conditions by increasing wetting and removal of surface layers formed by chemical products (Fleischer *et al* 1975). However, in the initial stages of etching, it decreases the wetting and causes reduction in the convective transport of the etchant from the surface of the filter to the bulk of the etchant.

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